STATE OF CALIFORNIA
DEPARTMENT OF PUBLIC WORKS
DIVISION OF HIGHWAYS

Summary of Discussion
on

CONTROL OF GYPSUM IN PORTLAND CEMENT BY THE EXPANSION-CONTRACTION TEST

at
Conference held at Fresno, California
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This conference was proposed by California Division of Highways, Materials and Research Department, for the purpose of discussing the technical aspects of controlling gypsum in portland cement at its optimum value as determined by the expansion-contraction test of Ottawa sand mortars. Data obtained in co-operative testing between the cement mills of California and the laboratory of the Division of Highways provided the basis of the discussion.

The meeting was attended by the following representatives:

J. W. Gilliland	Ideal Cement Company
R. M. Durland	Calaveras Cement Company
Robert Layton	" " "
J. R. Romig	California Portland Cement Co.
R. A. Loveland	" " " "
O. L. McCain	" " " "
R. A. Schott	" " " "
F. Norman Jones	Pacific Cement & Aggregates, Inc.
O. E. Jack	Permanente Cement Company
R. E. Sharpe	" " "
E. L. McMaster	" " "
E. A. Curley	Riverside Cement Company
W. N. Kopenhefer	" " "
L. L. Cook	" " "

J. B. Alexander	Southwestern Portland Cement Co.
L. R. Indermuehle	" " " "
A. R. Bryan	Monolith Portland Cement Company
John Partlow	" " " "
Bailey Tremper	California Division of Highways
W. E. Haskell	" " " "

Each cement mill in the State except that of Blue Diamond Corporation was represented. Among the remaining twelve mills, the chief chemist of each was present except those of Ideal and Calaveras.

Bailey Tremper acted as Chairman of the meeting.

Need of Control of Gypsum

The chairman opened the meeting with a presentation of the need of minimum volume change in concrete. Views of concrete that have deteriorated seriously by reason of excessive drying shrinkage were shown. It was pointed out that the use of cement that was deficient in gypsum undoubtedly played a major part in the distress. Slides were shown depicting the curling of pavement slabs and the effect of drying shrinkage in reducing resistance to traffic loads.

Equipment and Supplies Used in Test

The chairman stated that although the results obtained in co-operative expansion-contraction tests were in fairly good agreement, consideration of means of improving the test was still desirable. It was brought out that all mill

laboratories used the plywood drying cabinet (specified as alternate equipment in the test method), whereas the Division of Highways used a room accurately controlled for temperature and relative humidity. It was brought out that several laboratories had experienced trouble with the motor of the fan burning out and that replacement had been made with a different type of motor. One mill used reagent grade of calcium sulfate; the remaining mills prepared pulverized gypsum by grinding in the laboratory. California Division of Highways used a grade of gypsum marketed as Terra Alba.

Interpretation of Test Data

The chairman reviewed the use of a parabolic equation in estimating the relationship of SO_3 in cement to its optimum value. The results of the co-operative tests had been calculated on the assumption that a coefficient of 0.018 in the equation, while not exactly correct for all of the cements, was close enough to the true value to be usable.

Review of Co-operative Test Data

The chairman pointed out that when expressed in terms of the departure of SO_3 content of the test samples from their optimum value, the co-operating laboratories obtained results which in the main were within 0.25 percent relative to optimum. It appeared therefore, that if cement were furnished with SO_3

within 0.25 percent of optimum, as shown by the expansion-contraction test, other laboratories using the same test should also obtain results within ± 0.25 percent of optimum.

The tabulations in the test report showing the indicated optimum SO_3 percentage for each sample were discussed at considerable length. The samples from some mills indicated a rather constant optimum SO_3 content throughout. In other mills a few of the samples had indicated optimum values that were at serious divergence from the remainder. Many of these divergences were explained by known differences in the characteristics of the cement in the sample such as exceptionally fine grinding, laboratory rather than mill grinding, differences in chemical composition, etc. Some of the divergences could not be thus explained. For example, one of the samples from mill J had an exceptionally low indicated optimum SO_3 content. This cement had been in storage for 8 months and indicated the possiblity that the value for optimum had changed during storage. If this should prove to be the case, it would be a severe handicap in the production of cement with optimum SO_3 at the time of shipment. A number of representatives agreed to retest samples that had been in storage for some months since the original test, to determine if a change in optimum had occurred.

The samples from several mills showed large and unexplainable divergences from indicated optimum among the samples tested. For example, among 13 samples submitted from mill H,

about half had indicated optimum values of about 1.25 percent SO_3 and others were in the neighborhood of 1.75 percent. The mill chemists stated that all samples were taken from regular mill production and as far as known, were similar. This mill agreed to grind clinker with a number of steps in SO_3 and to test and submit samples for stepwise additions of gypsum in the laboratory for determination of true optimum as well as indicated optimum by the "two-step" expansion-contraction test.

One of the chemists stated that with his cement, the highest strength had been obtained when the SO_3 was lower than the optimum as shown by the expansion-contraction test. The chairman stated that this finding was not in accordance with the results of many carefully controlled tests and suggested further investigation with the cement in question.

Mill Grinding vs. Laboratory Addition of Gypsum

The chairman stated that the question had been raised as to whether the optimum obtained by mill grinding with the full amount of gypsum gave different results than were obtained by laboratory addition of pulverized gypsum to a cement. None of the participants offered data to show what the effect, if any, might be. However, one chemist expressed the opinion that there was a difference.

Determination of Proper Value of "p" for Each Cement

The chairman stated the value of 0.018 for "p" in the equation used in computing optimum SO3 from routine contraction tests had been selected as an average of the data submitted at the time. Subsequently data from one mill indicated that the value for that cement should be about 0.028, which value would make considerable difference in the value for indicated optimum as computed from "two-step" test results. Such a coefficient would indicate a much sharper response to variations in SO3 and would require control of gypsum within narrower limits for minimum contraction. It was pointed out that a great deal more data that would permit the determination of "p" would be required by most of the mills. It was felt that the mills had not done sufficient work on this factor.

Relative Cost of Gypsum and Clinker

In reply to a question by the chairman as to whether there was sufficient difference between the cost of gypsum and clinker at the mill to make a significant difference in the cost of finished cement, one chemist replied that the cost to that mill for gypsum was about 1.7 times that of the clinker. The general consesus however, was that the difference in cost was too small to be of economic consequence compared to the benefits to be derived from the addition of the optimum amount of gypsum.

Problems in Mill Proportioning of Gypsum

The statement was made that with existing facilities for proportioning gypsum and clinker, the desired SO_3 content could not consistently be secured within a tolerance less than $\pm$ 0.2 percent. It was stated that although clinker and gypsum were fed to the grinding equipment on belts, the feed was adjusted volumetrically. The chemists from the other mills did not disagree with above value of obtainable precision.

Circumstances under which it might be desirable to produce cement with SO_3 content differing from optimum

Partlow of Monolith stated that his cement containing optimum SO_3 could conceivably have an undesirable long setting time which would react unfavorably on selling problems.

Curley of Riverside spoke of problems of carrying separate supplies of cement in stock if Division of Highways' specifications were different from that of other producers. Such a situation could arise if the SO_3 content for optimum were above or even slightly below the maximum permitted under ASTM and Federal specifications.

Gilliland of Ideal expressed fear that optimum SO_3 might produce false set particularly with Type III cement. Schott of California Portland Cement Company expressed a similar idea.

Romig of California Portland Cement Company stated that the natural reaction to any evidence of false set would

be reduce the SO3 content without regard to its optimum value.

Partlow of Monolith stated that the optimum for his cement evidently is about 2.35 percent and that this value is too close to the presently specified maximum of 2.50 percent for Type II cement to be workable. The tendency would be to work toward the lower end of a proposed range of ± 0.25 percent of optimum.

Is control of SO3 by the expansion-contraction test technically feasible? If so, within what limits?

In introducing this subject, the chairman emphasized that he was asking for statements based purely on technical considerations without regard to management or policy considerations.

Gilliland of Ideal inquired if every sample would be required to pass the test. The chairman replied that if incorporated as a specification requirement, that would be the case.

Durland of Calaveras was of the opinion that more test data was necessary and should be obtained from all phases of manufacturing.

Romig of California Portland Cement Company called attention to the considerable number of unexplainable "out-of line" results in the co-operative tests notwithstanding

reasonably good agreement between two laboratories. He questioned the ability of the expansion-contraction to definitely indicate the optimum SO_3 in all cases and that anomolous results if obtained, would react to the disadvantage of the purchaser as well as the producer. He also stated that the 7-day elapsed time in completing the test was a great disadvantage.

Jones of PCA believed that Santa Cruz Type II cement could be manufactured within prescribed limits as determined by the expansion-contraction test without technical difficulty.

Curley and Cook of Riverside believed that cements in their two mills could be manufactured to meet the test requirement without technical difficulties.

Alexander of Southwestern believed that no difficulty would be encountered in his mill but stressed the need of more test data.

Partlow of Monolith again referred to difficulties arising from the fact that the optimum for his cement was so close to specification limits.

Gilliland of Ideal said that the test method must unmistakably indicate the true optimum for the cement in order to make it applicable to individual samples.

Jack of Permanente expressed the belief that considerably more work must be done before the method could be considered to be workable as a method of control. McMaster of Permanente

inquired what the penalty would be, if after adoption of a requirement based on the test method, the mill failed to furnish cement that conformed. The chairman replied that if the specifiactions were so written, cement that did not conform would be subject to rejection. The chairman stated however, that at this point he was not proposing a specification requirement based on the test. The object of the inquiry was to learn of anticipated technical difficulties in staying within limits of $\pm$ 0.25 percent of optimum if the test method were used.

A number of chemists referred to substantial quantities of SO_3 in the clinker resulting from the use of sulfur bearing fuels such as oil. It was pointed out that these sulfates probably are combined with alkalies rather than calcium oxide and thus probably do not perform in the same way as gypsum. Gilliland of Ideal pointed out that the clinker sulfates should dissolve quickly and when dissolved, should be present as ions and therefore act in the same manner as gypsum.

Partlow of Monolith spoke of possible effects of mill temperature during grinding on variations in optimum SO_3.

The chairman referred to the fact that although in the main there was good agreement between the Division of Highways and the cement mills on the percentage of SO_3 in the co-operative test samples, there were a number of glaring exceptions in which the reported values differed by from 0.10 to over 0.20 percent. He inquired why this relatively simple determination

could not be reproduced more exactly. One of the mill chemists referred to the need of prompt results and said that frequently the period of digestion of the precipitate, which is specified to be from 12 to 24 hours, was shortened in mill practice and that the shorter period of digestion was known to give results that were somewhat low.

What further experimental work is needed?

This subject has been mentioned frequently under other headings. The chairman referred to the need of refinements in the test method itself and asked for volunteers to do such work. There were no volunteers.

Closing statements

The chairman stated that although the expansion-contraction test appeared to be adequate for the purpose, there was no intention to closing the door to other tests. He brought up the possibility of specifying a maximum value of contraction without regard to SO_3 content and its relationship to optimum. It was pointed out that when California Type II, low-alkali cements contained optimum SO_3, there was very little difference between brands in the values of measured contraction. It was also pointed out that the successful use of such a requirement would necessitate that each laboratory be equipped

with accurately controlled temperature and relative humidity space, that the presently used plywood drying cabinets would not be adequate. Representative values of contraction could be selected from the test data of the Division of Highways which has used a well controlled room for the contraction tests. A number of the mill chemists stated that such a measure would not eliminate any of the mill problems that are brought about by control by the expansion-contraction test.

The chairman again emphasized the importance in highway work or producing concrete of minimum volume change and stated that adequate control of gypsum in portland cement was an important part of the problem. He stated that 15 years have elapsed since Lerch first published his findings on the effect of gypsum and during that period industry has done little toward putting the principles into effect. He warned those in attendance that consumers might at any time take it upon themselves to write requirements into specifications that were aimed at securing cement with gypsum in optimum amount. For this reason it would be well for each producer to devote more time in investigating means of accomplishing such an objective to the end that they would be well prepared in the event of specification requirements for cement with optimum gypsum.

The chairman thanked those in attendance for the time spent in attending the meeting and for their courteous discussion of the problems involved.